

CHROM. 5533

The separation of the Gold(III)-Rhodamine B chloro complex from Fe(III), Sb(V) and Ga(III)-Rhodamine B chloro complexes by thin-layer chromatography

Cations such as Fe^{3+} , Ga^{3+} , Sb^{5+} , Au^{3+} and Tl^{3+} , in hydrochloric acid solutions with Rhodamine B, form intensely coloured Rhodamine B chloro complexes which can be extracted with organic solvents (benzene, diisopropyl ether, etc.). These complexes are used for the photometric determination of the elements mentioned, and since they are extracted under similar conditions and have similar colours, they mutually interfere¹. It might be interesting if a thin-layer chromatography (TLC) method could be employed for the identification of the complexes and analysis of their mixtures. For this purpose, the behaviour of Fe(III), Sb(V), Ga(III) and Au(III) Rhodamine B chloro complexes on silica gel and cellulose layers has been investigated

Experimental

Materials for layers. Microcrystalline cellulose for TLC and Silica Gel G (Merck). Aqueous suspensions of cellulose (cellulose:water = 1:3.5) and of silica gel (silica gel:water = 1:2.5) are coated on 10 × 15 cm glass plates. The cellulose layers are dried in air at room temperature, and the silica gel layers at 120° for 1 h.

Reagents. FeCl_3 , SbCl_3 , metallic Ga, metallic Au, Rhodamine B (all p.a.).

Procedure. The iron(III) and antimony(III) chlorides were dissolved in 6 M hydrochloric acid, and the antimony was oxidized with sodium nitrite. The gold and gallium were dissolved in a little *aqua regia*, and then diluted with 6 M hydrochloric acid. Rhodamine B solution (0.5% in 6 M HCl) was added to form the complexes, which were extracted with a mixture of benzene and ether (3:1). Small volumes of each extract and of a mixture of them were applied on the chromatoplates with glass capillaries. Development was carried out by the ascending technique, over a distance of 10 cm, in a 12 × 8 × 18 cm glass chamber at room temperature. The organic phase was used for development if the solvents were not completely miscible.

Results and discussion

Experiments have shown that the Rhodamine B chloro complexes of Fe(III), Sb(V) and Ga(III) behave very similarly on thin layers of cellulose and Silica Gel G. Differences exist between the R_F values on cellulose and silica gel in the same solvent system, but on the same layer and in the same solvent system the R_F values of all three elements are similar. Slightly different R_F values are sometimes obtained for the single spots, but mixtures cannot be separated. In several experiments, mixtures gave three spots, e.g. on cellulose with ether-8 M HCl (15:30) and benzene-ether-6 M HCl (30:10:10), but identification was not possible, since the R_F values of the single spots were very similar (usually 0-0.1) and were not always reproducible. These three Rhodamine B chloro complexes cannot be resolved by TLC.

The behaviour of the gold(III) complex is completely different, however, and it can be separated very easily from the mixtures on cellulose or Silica Gel G layers. On cellulose layers, gold(III) is separated fairly well from the mixture with benzene as solvent, but is completely separated with the solvent systems benzene-ether-HCl,

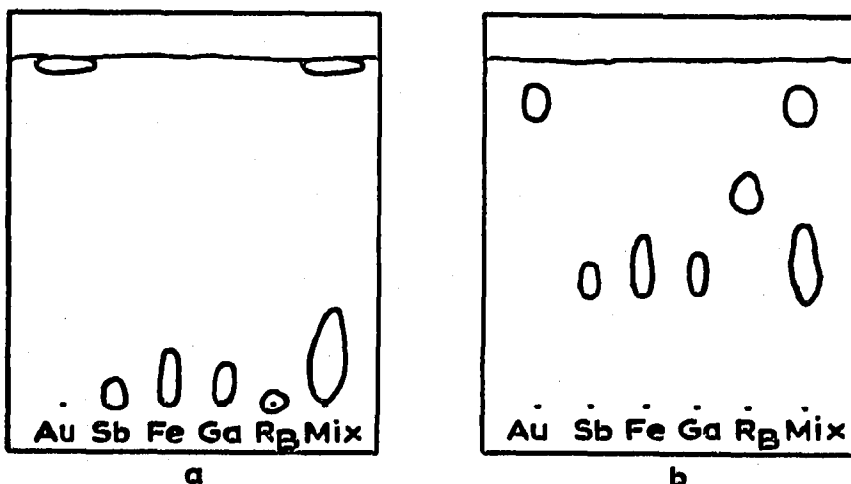


Fig. 1. Thin-layer chromatograms of Fe(III), Sb(V), Ga(III) and Au(III) Rhodamine B chloro complexes. (a) Layer, cellulose; solvent system, benzene-ether-8 *M* HCl (30:10:10). (b) Layer Silica Gel G; solvent: ethanol.

benzene-ethanol-HCl, ether-HCl, etc., especially benzene-ether-8 *M* HCl (30:10:10) and ether-8 *M* HCl (15:30), which give the gold(III) spot at the solvent front in a narrow band without a tail, while the spots of other elements remain near the start (Fig. 1a).

On silica gel layers, gold(III) is separated from the mixture by numerous solvents such as ethanol, acetone, ether-acetone, benzene-ether, ethanol-ether, benzene-ethanol, ether-HCl, ethanol-acetone, etc. With acetone and with ether-containing solvent systems (ether-acetone (10:10) and ether-8 *M* HCl (15:30)) the narrow gold(III) spot is at the solvent front, the other spots at or near the start (R_F values 0-0.15), while with ethanol and with benzene-containing solvents (ethanol-ether (20:5), ethanol-acetone (10:10), ether-benzene (30:10) and benzene-ethanol (40:5)), the almost round spots of gold(III) have R_F values from 0.85 to 0.95 whilst the other (elongated) spots have R_F values of about 0.3 (Fig. 1b).

In all experiments, Rhodamine B was also investigated and had R_F values of about zero, except with ethanol-containing solvent systems on Silica Gel G in which its R_F value was 0.5-0.6. Consequently Rhodamine B cannot affect the identification of the gold(III) spot.

The extracts of Ga(III) and Sb(V) Rhodamine B chloro complexes are red, and those of Fe(III) and Au(III) are red-violet, but on cellulose layers all the spots are red-violet; on Silica Gel G the gold(III) spot is red-violet, whilst the others (and Rhodamine B) are light red. In this manner it is possible to identify the presence of the gold(III) Rhodamine B chloro complex on silica gel very easily.

Department of Analytical Chemistry,
Technological Faculty, University of Zagreb,
Zagreb (Yugoslavia)

Z. ŠOLJIĆ

1 G. CHARLOT, *Les Méthodes de la Chimie Analytique, Analyse Quantitative Minérale*, 5ème Éd., Masson et Cie., Paris, 1966, pp. 591, 758, 825 and 941.

Received July 7th, 1971